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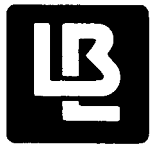

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CROSS-SECTIONAL TEM SPECIMENS FROM METAL-CERAMIC COMPOSITES †

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The demand for characterization of metal-ceramic composites has led to a number of TEM investigations of the microstructure and chemistry in the vicinity of the metal-ceramic interface. The atomic arrangements at such interfaces are of scientific as well as technological interest because they essentialy determine the mechanical properties of the composites. The accuracy of the results obtained using analytical as well as high resolution electron microscopy will be affected by changes in specimen thickness across the interface. In our efforts to characterize the metal-ceramic interface in thermal barrier coatings (TBC'S) we noticed preferential ion-milling of the metal in conventionally prepared (ground down to 100 μm, mechanically dimpled to 30 μm thickness and then ion-milled to perforation) TEM samples and hence it was impossible to get uniformly thin areas near the interface. The sample preparation technique described earlier by Bravman et al (1984) was modified so that samples of uniform thickness at the interface in thermal barrier coatings as well as at nickel particles dispersed in a zirconia matrix, could be obtained. Only the modifications in the technique are described here.

Materials and Methods

The thermal barrier coating samples (courtesey A. Levy, Lawrence Berkeley Lab., CA) have rough, porous surfaces and a very fluid adhesive like "M-Bond 610"* is not effective in forming a uniform layer. "Devcon"* was found to be much better for this purpose. The one to one mixture of hardner and resin is quite viscous and hence does not penetrate all the pores in the ceramic coat. It is then possible to obtain a thin uniform layer of this adhesive between two ceramic faces of TBC'S. The two pieces were clamped in a vice similar to the one described by Bravman et al (1984), and the adhesive was cured at 70°C for 8 hours. Slices 200 μm thick were cut from the cured sample and three millimeter discs with the interface in the center were cut using slurry cutter*. These discs were ground down to ~ 120 μm thickness on 600 grit paper and then polished on Al_2O_3 paper (0,00,000, and 0000). Final polishing with an activated final polish solution* produced scratch free surfaces. This is necessary to obtain uniform ion milling. The samples with nickel particles dispersed in zirconia obtained by hot pressing were sliced (using diamond saw*.) and 3mm discs cut with a slurry cutter*. These specimens also were ground down to about 120 μm thickness and one face polished as described above.

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* 'M Bond 610 Adhesive': Measurements Group, Raleigh, NC 27611
'Devcon 2 Ton Epoxy': Devcon Corporation, Danvers, MA 01923
'Slurry Cutter', 'Diamond Saw': South Bay Tech. Inc. Temple City, CA 91780
'Colloidal Silica Polishing Solution, Buehler LTD., Lake Bluff, IL 60044

All the samples were then dimpled from the unpolished surface using a dimpler*. The dimpling procedure for these metal-ceramic composites differs from that of Bravman et al (1984). It is essential to reduce the ion milling time to an absolute minimum to avoid developing a step at the metal ceramic interface. We therefore use very careful dimpling to obtain a small hole ($<$ 0.5mm in diameter). It is necessary to measure the thickness of the sample quite accurately. Care is also taken during mounting of the sample onto the dimpler stage so that the layer of adhesive is uniform beneathe the sample. The micrometer on the dimpler used to control the dimple depth is then set to 20 μm less than this thickness. The last 20 μm's are dimpled μm's at a time. It is thus possible to obtain a tiny hole at the center of the specimen if the dimpling tool is properly aligned and the load and dimpling speed are so chosen that the dimpler arm is vibration free. We suggest a low dimpling speed (setting 3-4, VCR dimpler). Once a hole is obtained, the dimple is then polished to a mirror finish using the dimple polishing tool and syton. This also should be done vibration free by ensuring proper mounting of the polishing felt on to the tool. If proper care is taken electron transparent areas are obtained near the edge of the hole even at this stage. Otherwise, 1 or 2 hour ion-milling at lower voltages (4 to 5kV) and low angles, 15°, produce good TEM samples. Fig. 1 shows a typical dimpled and ion milled sample. A typical electron micrograph taken on one of these samples, Fig. 2, indicates uniformity of thickness across the metal-metal oxide-ceramic interface and illustrates the usefulness of this technique.

*Dimpler Model D200: VCR Group, San Francisco, CA 94312

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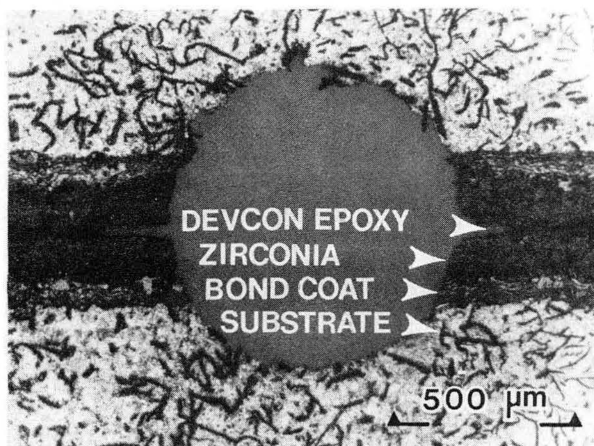

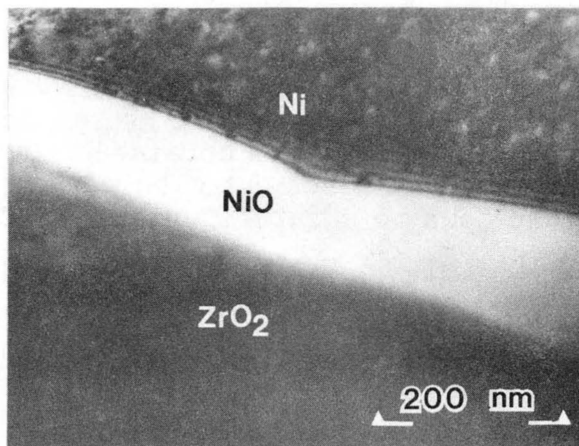

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Fig. 1

TEM sample from TBC'S after dimpling and 1.5 hr ion-milling.

Fig. 2

Bright-field image of interface between Ni, NiO and ZrO_2.

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